Local polarization reversal in KTP single crystals

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Single crystals of potassium titanyl phosphate ($KTiOPO_4$, KTP) with periodical ferroelectric domain structure are widely used for nonlinear optical applications including forward and backward second harmonic generation and optical parametric oscillation [1-3]. The domain structure evolution in KTP can be considered as a model process for investigation of the kinetics of the first order phase transitions in the crystals with C_{2v} symmetry. Previously, domain kinetics was studied *in situ* by optical methods [4,5], but, to our knowledge, local polarization reversal in crystals of KTP family was not investigated systematically [6]. Usage of the scanning probe microscopy methods gives the opportunity to study the domain kinetics with nanoscale resolution, which is very important for understanding the mechanisms of domain evolution.

We have studied KTP single crystals (Crystals of Siberia Ltd., Russia) grown by top-seeded solution method. The sample cut perpendicular to the polar axis was stuck to the ceramic plate on the UF-glue and polished down to 10 μm. The bottom electrical contact was provided by the indium tin oxide (ITO) electrodes deposited on the bottom side of the sample and the ceramic surface. The bottom electrode was grounded during switching and domain visualization.

Investigation of the local polarization reversal was performed by the scanning probe microscope NTEGRA Aura (NT-MDT). All experiments were performed at RT. The controlled relative humidity in the microscope camera ranged from 4% to 30%. The constant electric field pulse was applied to the top z+ polar surface by the conductive tip NSC14-Pt (MikroMasch®) with typical resonance frequency 160 kHz and force constant 5 N/m. Domain size dependences on pulse duration and amplitude were measured in the wide range: amplitude from 30 to 200 V (Fig. 1), duration from 10 ms to 200 s (Fig. 2).

The field was switched off only after the tip withdrew the surface to eliminate the backswitching effect. Domains have been written in arrays at the distance of 3 μm to each other in order to avoid their interaction. The switched domains were visualized just after polarization reversal by the same tip in piezoresponse force microscopy (PFM) mode by applying at 20 kHz modulation voltage of 3 V rms.

The switched domains were of hexagonal shape elongated in Y direction determined by the crystal symmetry. The obtained linear dependence of the domain length on the applied voltage (Fig. 1c) is typical for switching in uniaxial ferroelectrics [7,8].

We propose the following scenario of domain growth for elongated domains formed in the inhomogeneous field of the conductive tip: (1) domain nucleation under the tip, (2) domain growth in width due to step generation at the side walls, (3) domains growth in length due to step generation at the vertices and kink motion.

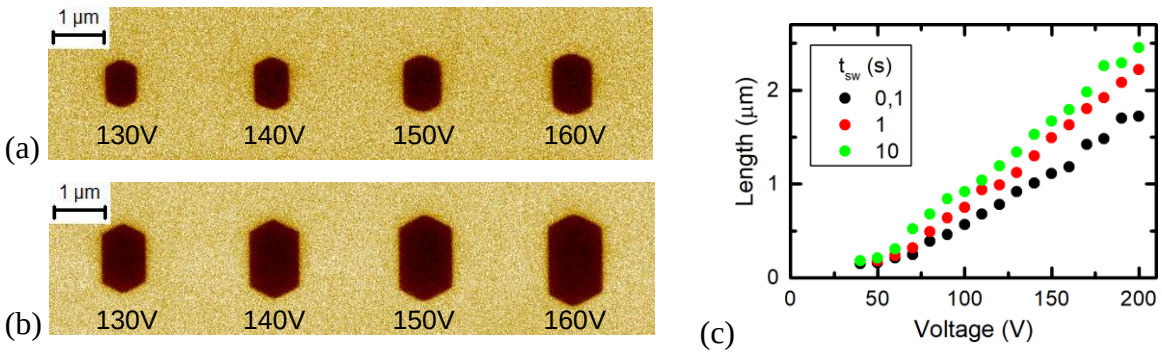

Figure 1. (a), (b) PFM images of domains obtained by local switching in KTP. Switching time: (a) 100 ms, (b) 10 s. Switching voltage: 130, 140, 150, and 160 V. (c) Domain length dependence on the pulse amplitude (switching voltage).

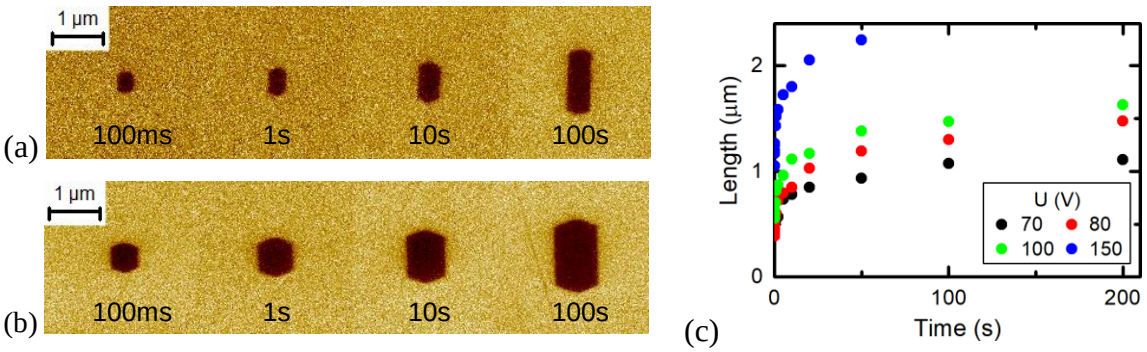

Figure 2. (a), (b) PFM images of domains obtained by local switching in KTP. Switching voltage: (a) 80 V, (b) 100 V. Switching time: 100 ms, 1 s, 10 s, 100 s. (c) Domain length dependence on the pulse duration (switching time).

The independence of the domain width on switching time for fields below 70 V has been attributed to the applied field value below the threshold for step generation at the domain walls. Domain length and width for pulse durations 100 ms and 1 s practically coincide in wide field range for low humidity (RH = 4%). It means that the domain rather fast reaches the size limited by the spatial distribution of the switching field. In the case of low humidity, the surface is free of water resulting in absence of external screening.

The obtained experimental data have been discussed in terms of the universal kinetic approach taking into account the influence of the screening retardation effect [9,10]. The spatial distribution of the residual depolarization field and switching field at the domain wall was calculated by COMSOL. The effect of step generation at the polygon vertices has been confirmed.

The research was made possible in part by RFBR (grant 16-02-00724) and by President of Russian Federation grant for young scientists (Contract 14.Y30.17.2837-MK). The equipment of the Ural Center for Shared Use “Modern nanotechnology” Ural Federal University was used.

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